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Low Temperature Concentration of Skim Milk by Direct Freezing and Vacuum Evaporation[1]

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ABSTRACT

Skim milk was concentrated to approximately 20% TS by direct freezing and ice filtration. The concentrate was vacuum evaporated to 45% TS with the same equipment. Protein denaturation was minimal during concentration. Surface tension of milk samples was measured during concentration close to 0°C. The process cost for plants of various sizes were estimated from pilot plant data. The estimated cost and the good quality of the reconstituted skim milk support the recommendation that the process should undergo further development.

(**Key words**: freeze concentration, skim milk, cost, taste)

Abbreviation key: **DFC** = direct FC, **FC** = freeze concentration.

INTRODUCTION

When production exceeds immediate demand, skim milk is concentrated for short-term cold storage or converted to powder for longer term, unrefrigerated storage. Concentration is most often carried out in multistage evaporators; evaporating temperature for the first effect is 70°C. Parris et al. (15, 16) established that heating skim milk, beginning at about 70°C, causes irreversible aggregation of β-LG. Milk concentration using membranes does not cause thermal denaturation, and several studies of membrane concentration of milk have been reported by Garcia and Medina (5), Zall (22), and Olesen and Jensen (14). However, membranes are costly, and development continues.

Freeze concentration (FC), which has been studied for desalination of sea water, has low energy costs for large-scale use, as shown by Heist (7). The FC is also well suited to food liquids because, like membrane concentration, FC exposes the milk to negligible heat stress and less degradation by substrate circulation. In 1986, a cost-benefit analysis of milk FC to reduce transportation costs over long distances in Australia was reported by Langdon and Cox (11); their analysis indicated that transportation of ≥480 km was needed to make concentration cost effective. Over the last decade, a project has been underway to adapt the indirect FC process used for juices and dilute beverages to milk; progress has been reported by van Mil and Bouman (21) and Swientek (20). Promising results from indirect FC pilot plant tests have recently been cited by Buss (3), laboratory studies described by Hartel and Espinel (6), and improvements suggested by H. G. Schwartzberg at the 1988 summer meeting of the American Institute of Chemical Engineers (unpublished). Indirect FC processes, although they certainly prevent overheating, have the potential to damage fluid constituents that are sensitive to supercooling.

To determine the practical feasibility of concentrating milk by a method that avoids thermal denaturation, a developmental investigation of direct FC (DFC) was initiated, starting with a pilot plant provided by the US Department of Energy (under Agreement Number EC-77-Mw01-1052; 1982). The latter part of that investigation, showing the technical feasibility of DFC, is reported here.

The latent heat of water is removed by DFC, resulting from freezing by evaporation of liquid water. The evaporation and freezing are driven by the low pressure maintained over the

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[1]Mention of brand or firm names does not constitute an endorsement by the USDA over others of a similar nature not mentioned.

slurry. In order to freeze and to evaporate simultaneously, the slurry must be maintained at a pressure below the triple point of the liquid, that is, below the pressure at which solid, vapor, and liquid are near equilibrium. Because of direct contact between the chilling and chilled components, which are evaporating and freezing water from the same solution, the DFC freezing rate is not dependent on heat transfer through the solid freezer wall and ice covering it, but by heat transfer within the freezing, agitated liquid.

This procedure is in contrast to indirect FC, in which the latent heat of freezing is removed by conduction through a metal surface that is maintained well below the freezing temperature of the slurry. The DFC takes place <1°C below the liquid triple point, thereby exposing chilled liquids directly to minimal supercooling. However, the main reason for limited use of the indirect FC is not overchilling, but high equipment cost. The high cost is inherent to the ice separation method used, which produces a pure ice product but requires large ice particles and slurry ripening in an ice crystal growth tank. The ice separation method is effective but requires rapid and precise control to maintain a sharp separation; therefore, the process is divided into stages, requiring separate ice separators for each stage.

For milk FC processes, the difficulty of separating the ice from the concentrated milk increases as concentration increases, mainly because of diminishing ice particle size, which increases the capillary pressure of the ice bed. Concentrated skim milk is more difficult to separate from ice particles than fruit juice because its viscosity rises very rapidly with TS, which has been discussed previously by Dickey and Craig (4). Skim milk concentrated at low temperature forms protein-lactose aggregates that obstruct filtration, beginning at about 20% milk TS. The aggregates interfere with filtration before the viscosity alone is detrimental to ice separation, as shown with tests we have made of ice slurries formed from carboxymethyl cellulose solutions with viscosities equal to or greater than that of concentrated skim milk ice slurries that cannot be filtered.

MATERIALS AND METHODS

DFC Process Equipment

Figure 1 is a schematic diagram of the equipment used to concentrate batches of up to 1140 L of skim milk. The original milk was concentrated from about 9% TS to 20% TS by DFC. In DFC, water evaporates and chills the milk. Water vapor is mostly removed by condensation on a wetted wall above the agitated solution; small amounts of vapor and air that had been dissolved in the milk are taken out by a vacuum pump. Evaporation of the water drives a chilling process that freezes water in the agitated liquid if the water vapor and air removal processes maintain the pressure below the triple point of the milk. The triple point of fresh skim milk is only slightly below that of water, .61 kPa, but decreases to about .53 kPa at 20% TS. The milk is concentrated by recirculation through a loop containing the units for freezing and ice filtration until the filtration of the ice slurry becomes impractical. The concentration time of this batch process depends on the initial mass of fresh milk being concentrated.

Figure 1 shows the central vessel, a 61-cm diameter stainless steel cylinder divided into 1.27-m upper (condensation) and .66-m lower (evaporation) sections by a horizontal pan. The pan has a central 18-cm diameter rimmed opening, which allows passage of the low pressure water vapor but retains a shallow pool of liquid. A saturated salt solution is sprayed onto the upper end of the wall of the upper section, draining as a vertical film an area of approximately 1.5 m^2 to the pan at the bottom. Water condensed on the falling film drains, with the salt solution, through a port above the pan. An automatic control drains salt solution when the recirculating mass exceeds the original charge of 43 L. Drained solution was continuously weighed, and the condensation rate was calculated from the drained weight measurements. All measurements, including temperature, pressure, and flow rates, were automatically logged every 20 s.

The heat of water vapor condensation is transferred through the salt solution and vessel walls to a jacket around the upper section, formed of coiled copper tubing. For this vessel, heat transfer through the condensation section wall controls the overall condensation and, consequently, the rates of evaporation and freezing.

The lower (evaporation) section of the vessel contains inlets, outlets, a viewing port, and sanitary connections for measurement of temperature and pressure. Two vertical baffles (76 × 23 cm) are attached to the wall on a diameter 5 cm from the agitator, which is centrally mounted on the bottom of the tank. Milk freezes rapidly at the triple point and adheres to smooth vertical surfaces if they are not vigorously rinsed. Agitation must exceed 200 rpm to provide sufficient rinsing to keep ice from accumulating on the walls and feed spray nozzle.

The freezer is connected through inlet and outlet ports to sanitary progressing cavity pumps (1FG3 Moyno®; Robbins & Myers Inc., Springfield, OH), which convey milk and seal the low pressure evaporation section. Rates of inlet and outlet flow are metered, and the feed rate is controlled by a pressure difference between the condensation section and the bottom

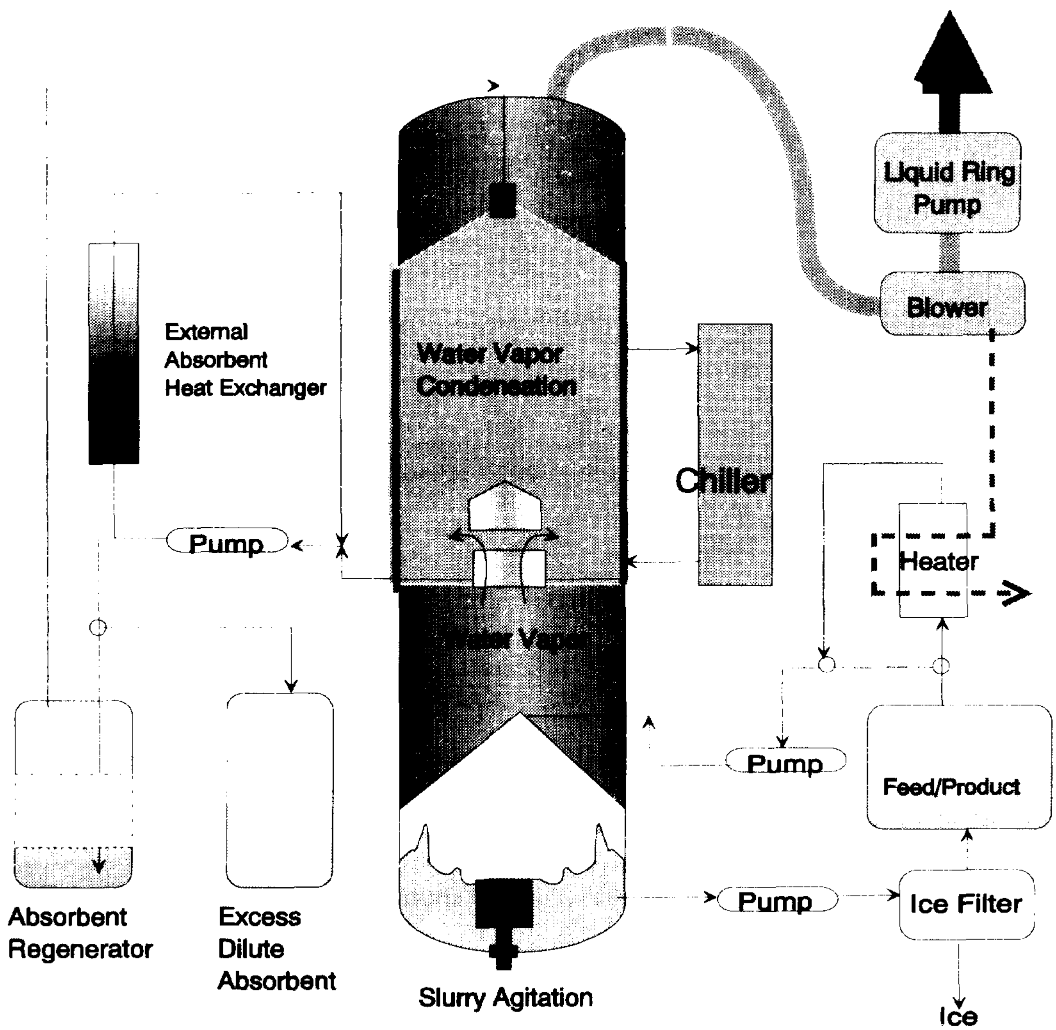

Figure 1. A schematic diagram of the direct freeze concentration process.

of the evaporation section (at the agitated ice slurry outlet). This control method maintains the fluid mass in the freezer near 45 kg.

Ice is separated from the slurry by pumping through a horizontal, 25 cm wide × 1.2 cm high, slotted rectangular channel. Concentrated milk drains through the slots at the inlet end of the channel, driven by the slurry pressure. At the ice outlet end of the channel, milk drains from the consolidated, moving ice sheet into vacuum boxes. Rinse water is applied to the ice sheet upstream of the vacuum boxes at the outlet end. As the milk concentration increases, more of the filtrate is collected from the ice outlet end of the filter. The quantity of TS lost with the ice can be reduced by increasing the flow of rinse water; however, this increase reduces the DFC rate because some of the rinse water is recirculated with the milk that is recovered by the vacuum boxes. The filter is clamped together and can be disassembled for cleaning and sterilization between runs.

Skim Milk Characterization

Milk used in these tests was Grade A pasteurized and homogenized skim milk from local dairies and was labeled as lots A, B, and C. Specific gravity and viscosity of the milk fed to the freezer, and temperature of the milk and ice slurry, were measured with a flow-through, vibrating tube type instrument (Dynatrol®; Automation Products, Houston, TX) and a piston in-line viscosity sensor (Cambridge Applied Systems, Inc., Cambridge, MA); measurements were electronically recorded at 20-s intervals on a VAX model 4300 minicomputer (Digital Equipment Corporation, Nashua, NH). Surface tension, TS, and lactose contents of hourly filtrate samples were measured after the concentration was completed. Surface tension was measured using the technique of maximum bubble pressure with a resolution of ± .1 Mn/m (Sensa-Dyne® Model 6000 Bubble Tensiometer; Chem-Dyne Research Corp., Mesa, AZ). The TS content was determined by weighing before and after heating over a steam bath and by vacuum drying. The heating ensured that no solids were lost in the subsequent vacuum drying step, which was carried out overnight. Lactose content was determined by HPLC after precipitation of protein and fat, following the method of Kwak and Jeon (10).

TABLE 1. Initial and final operating conditions for the pilot plant.

Milk flow rate to freezer, kg/h	454
Water evaporation rate	
Freezing, kg/h	12.7
Nonfreezing, kg/h	15.9
Freezer agitation rate, rpm	400
Pressure	
Freezing, kPa	.50 to .60
Nonfreezing, kPa	.62 to .68

Microbiological Testing

The microbiological quality of milk before and after concentration, but before frozen storage, was determined by standard methods for spiral plate and coliform counts (13, 17). All samples were within acceptable limits (<30,000 total cfu/g and <10 cfu/g of coliforms).

Sensory Evaluation

Skim milks, concentrated to 20 to 45% TS for tasting, were stored frozen at − 17°C for 15 d before thawing for 72 h at refrigerated temperature (4.4°C); an unconcentrated control milk was treated similarly. After thawing, the concentrates were reconstituted to 9% TS with spring water (Great Bear Spring Co., Teterboro, NJ) and stored overnight at 4.4°C. Prior to tasting, the reconstituted 45% concentrate was gently heated to 54.4°C, immediately chilled to 4.4°C in an ice bath, and stored overnight at that temperature. This procedure was necessary to ensure that all lactose crystals had redissolved.

Samples were rated for preference on the nine-point scale of Peryam and Pilgrim (18); coded samples were served at refrigerator temperature (4.4°C) in booths under colored lights to eliminate bias that was due to slight color variations. Judges consisted of >50 staff members of the eastern Regional Research Center, some of whom were experienced in tasting a variety of food products. Statistical evaluations for significance were by ANOVA and Duncan's multiple range test (1).

RESULTS

Characterization of Skim Milk

Table 1 lists the essentially steady operating conditions for a typical FC run of milk C. The

TABLE 2. Properties of initial skim milk.

Milk	Date	TS	Protein	Lactose	Surface tension[1]
		(%)			(mN/m)
A	Aug 23	9.49	4.89	4.97	71.8
B	Sep 27	8.95	4.57	4.56	68.2
C	Oct 26	9.07	*	4.11	64.8

[1]Measured at 0°C.

*Data missing.

pressure dropped slightly as the activity of the milk declined with increasing concentration; this effect held for FC and vacuum evaporation in the pilot equipment because the rates of both were limited by the water vapor condensation rate. The variation of surface tension, at 0°C, of three lots of milk concentrated in 1993 is shown in Table 2. Interestingly, the surface tension of the fresh milk at 0°C varied directly with measured lactose content. The surface tension variation among milk lots, as delivered, was greater than the variation over the range of concentration achievable by DFC (Figure 2). The variations of milk specific gravity and viscosity are shown in Figure 3. These measurements were taken on the stream flowing to the freezer. Viscosity measurements were made at 20°C and specific gravity measurements at 2°C, until 140 min, when freeze concentration was stopped. At 180 min, vacuum evaporation without freezing began, and the specific gravity measuring system temperature rose to 16°C. The specific gravity of milk reached the maximum value measurable by the system (as calibrated) at 265 min.

Sensory Evaluation

The mean acceptance ratings were 6.32 (SD = 1.81) for the control, 6.62 (SD = 1.40) for the reconstituted 20% TS concentrate, and 5.74 (SD = 1.88) for the reconstituted 45% TS concentrate. Mean scores for the control and the 20% TS concentrate fell between "like slightly" and "like moderately" with the rating system used; the score for the 45% TS concentrate fell between "neither like nor dislike" and "like slightly". Statistical analysis showed that the 20% TS concentrate was significantly ($P \leq .01$) different from the 45% TS concentrate but was not significantly different from the control; the 45% TS concentrate was also not

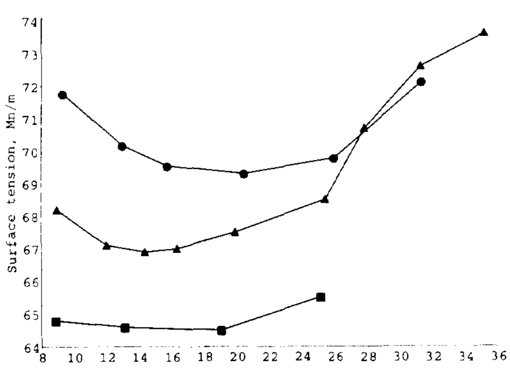

Figure 2. Surface tension at 0°C of skim milk A (●), skim milk B (▲), and skim milk C (■) with concentration.

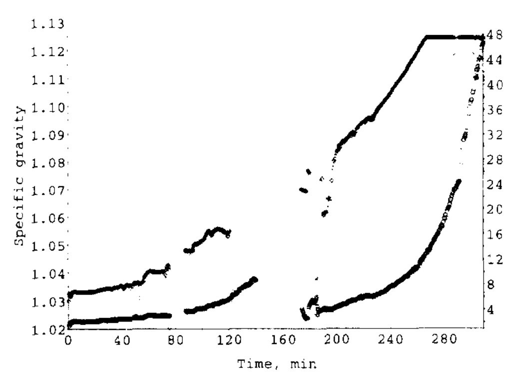

Figure 3. Skim milk specific gravity (·) and viscosity (o) changes with direct freeze concentration and vacuum evaporation. Freeze concentration stops at 140 min, and vacuum evaporation begins at 180 min.

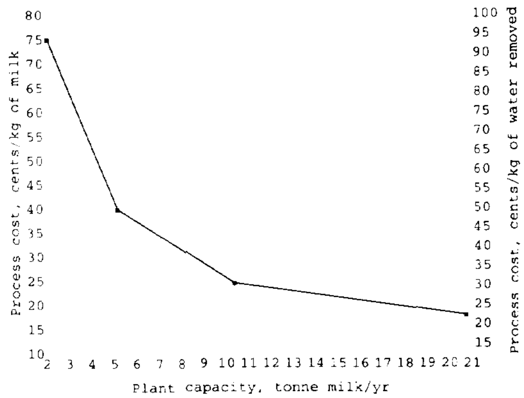

Figure 4. Effect of facility scale on freeze concentration and vacuum evaporation cost –19% TS skim milk concentrated to 45% TS. The left ordinate is cost per kilogram of 9% TS skim milk, and the right ordinate is the cost per kilogram of water removed from the milk.

significantly ($P \leq .01$) different from the control. Results were the same as those from a preliminary trial conducted with the same samples and fewer judges. These results are similar to previous hedonic tests of skim milk; if future sensory evaluation of skim milk quality are made, panels should be drawn from a population familiar with the taste of skim milk.

Cost Estimate of the Process

The cost of DFC and vacuum evaporation was estimated for plants of various sizes. Those costs, based on our pilot plant experiences, are shown in Figure 4. Plant labor was the major operating cost. The service of a full-time plant employee to oversee the plant operation and provide maintenance was included at an all-inclusive rate of $20.00/h for the 16 h/d that the facility operated and the 1 h/d required for cleaning. The labor costs per unit of production decreased as the capacity of the facility increased and could be reduced to levels lower than those we projected if other, intermittent work was available for the employee to perform during steady operation. Physical plant costs were the second largest component of the skim milk DFC cost. The plant costs used in this study were based on the uninstalled costs of our pilot plant equipment. Sets of these are listed in Table 3.

TABLE 3. Pilot plant costs.

Description	1994 Cost
	($\$ \times 10^3$)
Controls and instrumentation	40
Freezer-condenser	32
Chiller, 7.5 kW	23
Vacuum unit	14
Feed tank, 1 m^3	9
Piping and valves	9
Ice separator	7
Pumps	6
Total	140

Installation and indirect costs to construct a complete facility were calculated from Lang estimation factors to obtain the capital cost of a commercial facility with a capacity of 209,000 kg of skim milk/yr. Capital costs of similar plants with different capacities were then developed using ratios that correlated plant cost and capacity. The costs of utilities, chemicals, and cleaning supplies were the third component of the process cost. Those costs were proportional to capacity and are shown in Table 4 for the pilot plant. Profits, overhead, and administrative costs were not included, nor was the cost of the skim milk.

DISCUSSION

To recover most of the milk from the filtration process, the ice bed formed by filtration must be rinsed. The success of rinsing generally decreases as pore size decreases with increased concentration of protein in the milk. Bomben et al. (2) proposed that, during freezing, solute accumulates around each ice particle, retarding flow of supercooled water to the freezing surface. Thus, ice particles grow and coalesce more slowly at higher solute concentration. We expect, but have not confirmed, that most of the finer ice particles are melted when the slurry is pumped from the freezer. Thus, the particle size of slurry ice depends on the backpressure controlled by the ice filter. The capillary pressure of the ice bed formed during filtration increases as the particle size, and, thus, pore size, decreases (9). At particle sizes <60 μ, the pressure rises sharply as size decreases, preventing further removal of milk by air (or water) rinsing. Ice filtration at in-

TABLE 4. Operating consumption, pilot plant scale.

Consumable	Use rate	Unit cost	Cost
	(/h)	($/unit)	($/h)
Electric power, kW	19.5	.05	.97
NaCl, kg	3.2	.22[1]	.70
Milk lost, kg	4.5	.33	1.50
Cleaning chemicals,[2] use	1	26	1.70
Total			5.85

[1]An upper limit, overflow absorbent could be concentrated to saturation using waste heat from the process.

[2]Two liters of Oakite® 31 and 4 kg of Chloro-tergent® (Oakite Products, Inc., Berkeley Heights, NJ) per cleaning, used once each 16 h.

creasing milk concentration may also be limited by precipitates fouling the filter. Lactose solubility is only around 10% at 0°C, corresponding to 20 to 25% TS of skim milk, but lactose can be maintained as a supersaturated solution to much higher concentrations. The ability to filter ice from slurries of skim milk concentrate at >20% TS seems to be unreliable with our present process. To concentrate skim milk to >20 to 25% TS, the evaporation pressure can be raised above the triple point by heating the feed to the freezer and by removing water from the milk as a low pressure vapor. Nonfreezing, nondenaturing evaporation can be carried out above the concentrate triple point using the same equipment as for DFC with no process interruption. The capability to continue concentration by vacuum evaporation beyond the composition at which FC is attractive allows the system to handle feed with various consistencies and prepare various products with relatively low capital cost. The rate of water removal without ice formation is much lower than with ice removal if the evaporation temperature is increased only slightly above the triple point, approximately one-seventh of the DFC rate (it would be one-eighth if there were no heat gain through the equipment). However, rates of condensation and, consequently, evaporation increase as pressure and temperature increase; the rate of 9 kg/h used to calculate the cost estimate for the 45% concentrate is the minimum rate for near triple point evaporation, resulting in a conservative cost estimate.

Skim milk can be vacuum evaporated at temperatures up to 70°C without appreciable thermal denaturation (16), and, because of the protective effect of increased TS compared with that of unconcentrated milk, DFC may possibly be evaporated at even higher temperatures without degradation, as suggested by Schmidt et al. (19). To determine the effect of increased evaporation temperature, hot tap water was fed to the freezer (acting simply as an evaporator), and the rate of water vapor condensation was measured for feed temperatures ≤64°C. Condensation rate, which was limited by the cooling capacity, reached 35 kg/h, and the rate of vapor removal through the pump increased from .1 to 1 kg/h, giving a total rate about four times that used for the preceding calculations.

Batch processing allows the farm or dairy to cope with a variable milk supply, minimizing inventory and avoiding the problem of restoring a continuous operation after process breaks. Equipment maintenance and cleanup can be carried out frequently without loss of operating time beyond that required by the cleaning or maintenance alone. Although no tests have been conducted, lactose hydrolysis apparently can be conveniently carried out during the vacuum evaporation step using a process such as that described by Iborra et al. (8).

To establish better the maximum temperature to evaporate FC skim milk without impairing concentrate quality, vacuum evaporation trials have to be made at higher temperatures than that reported in out study. The slightly lower acceptability for the 45% TS concentrate, although not significantly different from that of the control, may have been the result of slight flavor changes brought about by heating of the reconstituted sample to dissolve residual lactose crystals formed as a result of frozen storage. Neither the control nor the 20% TS concentrate was heated. Future trials with samples that have not been stored frozen are necessary to resolve this issue. Neither experimental sample was significantly different from the control (starting material) in consumer acceptance.

Future FC trials will provide an opportunity to identify correlations between the hedonic test and the appropriate objective physical properties. In anticipation of such tests, surface tensions of the skim milk concentrates were measured from a number of milks. Surface tension, which can now be measured accurately and easily with the method of maximum bubble pressure, may vary in milk concentrates with changes in hydrophobicity derived from processing. Hydrophobicity has been correlated with bitter taste in a number of foods (12) and may also be associated with undesirable flavors derived from conventional milk concentration. As shown in Figure 2, the measured surface tension of the concentrate varied more among skim milk batches than within any single batch. The surface tension for all batches decreased to a shallow minimum from 14 to 20% TS, depending on the skim milk.

CONCLUSIONS

The DFC costs and hedonic tests of skim milk C appear to justify testing the process at an appropriate site to determine commercial feasibility.

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